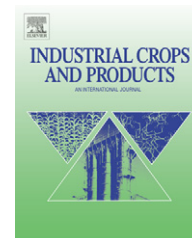


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Design properties for molded, corn-based DDGS-filled phenolic resin[☆]

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ABSTRACT

With the rapid growth in the ethanol fuel industry in recent years, considerable research is being devoted to maximizing the use of processing coproducts, such as distillers dried grains with solubles (DDGS), typically for livestock diets. Because these residues contain high fiber levels, they may be amendable to incorporation into polymers as well, which is an option that could garner greater economic returns. Thus, the goal of this study was to demonstrate the viability of using corn-based DDGS as a biofiller with phenolic resin, in order to produce a novel biomaterial. DDGS was blended with phenolic resin at four levels (0%, 25%, 50%, and 75%, by weight), and then compression molded at 13.8, 34.5, or 48.3 MPa (1.0, 2.5, or 3.5 tons/in.²) and 157, 174, or 191 °C (315, 345, or 375 F). Molded specimens were then tested for a variety of mechanical and physical properties. Pressure and temperature each had little effect on the resulting properties. DDGS, on the other hand, greatly influenced all of the properties. Tensile yield strengths ranged from 14.5 MPa (2102 psi) to 4.3 MPa (621 psi), while the Young's modulus ranged from 2296 MPa (333,000 psi) to 841 MPa (122,000 psi) as the DDGS content increased. For all time periods studied, water absorption increased as DDGS level increased. Moreover, as DDGS content increased to a maximum of 75%, biodegradability increased from 0% to 38% while the surface hardness decreased 25%. These results were similar to those from other studies that have investigated biofillers. Follow-up studies should aim to optimize the strength of the DDGS-blended resins through the use of coupling agents or other additives.

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1. Introduction

Currently, many plastic products utilize low-cost materials as fillers. Ideally the filler is added in a concentration that allows the product to retain sufficient mechanical strength, physical

properties, and final quality. Certain fillers may improve a plastic's other physical properties, such as thermal stability, color, and opacity. If the filler is added in a reasonable quantity, any degradation in strength from that of unfilled resin can be offset by the cost savings. Common fillers include clay, talc, ground

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limestone, carbon black, marble dust, glass, paper, wood flour, and metals, which are often added in concentrations ranging from 10% to 50% (by weight). To conserve petroleum resources and enhance biodegradability, bio-based fillers are receiving increased attention. Examples include wood flour, sugar cane, lignin, flax, grasses, bamboo, starch, chicken feathers, soy protein, and cellulose. This trend is consistent with recent United States governmental policies that prioritize the procurement of materials having significant bio-based content: this program is targeted to increase to a 50% bio-based level over the next several decades. Reviews documenting the use of biofillers in various plastics are available in the literature (Mohanty et al., 2000, 2001; Xanthos, 2005).

Phenolic resins are produced by chemically combining phenol and formaldehyde through a condensation reaction under alkaline conditions. Phenolic novolaks are designed to incorporate a curing agent, such as hexamethylenetetramine (hexa), and are referred to as two-stage systems; fillers and additives are then added to produce standard phenolic molding compounds, often employing wood flour as filler.

The dry grind and wet milling of corn to manufacture fuel ethanol is increasingly producing quantities of processing byproducts known as distillers grains. These materials are the non-fermentable residues left after the starch fraction of corn has been fermented with yeasts and enzymes to produce ethanol. Coproducts from dry milling include distillers dried grains (DDG), distillers dried grains with solubles (DDGS), condensed distillers solubles (CDS), and distillers wet grains (DWG), depending on specific drying and blending processes employed. The current use for all of these coproducts is in animal feeds. These distillers grains, especially the DDG and DDGS, represent potentially cost-effective biofillers, especially because they contain relatively high fiber content, sometimes up to 50% (dry basis). With the explosive growth of fuel ethanol production in the past several years, corn-based DDGS is now becoming generated in such quantities that some fear it may saturate the livestock feed market, and lose its value to the ethanol industry. Thus, it becomes attractive to search for other, higher-value uses for the DDGS. Accordingly, it is the intent of this study to report mechanical and physical design data for DDGS-filled phenolic plastic. To fully take advantage of the low-cost DDGS, the simple process involves mixing with phenolic powder to produce a molding compound which can then be reacted and cured through compression molding.

As only limited data exist in the literature for the combination of corn DDGS and phenolics (Tatara et al., 2007), it is instructive to review other studies that mixed biofillers with phenolic powder and produced compression molded test specimens. For example, Kharade and Kale (1998) studied a novolak-based molding compound which was produced by mixing phenolic, coconut shell powder, and other additives with wood flour and/or lignin; the inclusion levels of wood flour and lignin were systematically varied. Based on the total molding compound weight, wood flour/lignin percent ratios included 25.3/0 (i.e., no lignin), 15.3/10, 5.3/20.0, and 0/25.3 (i.e., no wood flour). Tensile strength and impact testing demonstrated greatly reduced performance as more lignin replaced the conventional wood flour: tensile strength decreased sharply, and at 0% wood flour (25.3% lignin) the decrease was 75%. Impact strength suffered similar decreases.

Along the same lines, wood flour and lignin (from Alpha grass) were examined in a phenolic (Hattali et al., 2005). Alpha grass lignin or wood flour constituted 0%, 5%, 15%, 25%, 35%, or 45% of the total mixture, with the remainder composed of phenolic and additives. Test specimens were molded at 20 MPa (1.5 tons/in.²) and 170 °C (338 F) for 17 min. As more phenolic was displaced, the impact strength decreased at approximately the same rate for either lignin or wood flour. Compared to no lignin or wood flour as fillers, the impact strength decreased about 40% and 35% for 25% lignin and 25% wood flour, respectively. For 45% lignin or wood flour, the impact strength was about half the strength without any of these biofillers.

Dried sugar cane pulp fiber served as another biofiller in a phenolic, hexa, and calcium stearate mixture, and was blended at 29% and 69% (Leite et al., 2004). Also, two different cane grain size distributions were tested: 35–80 mesh, and 80–170 mesh. The compression molding conditions were 14.7 MPa (1.1 tons/in.²) and 170 °C (338 F) for 10–15 min. In both cases, the addition of the sugar cane residue raised tensile strength significantly. For example, the 35–80 mesh (which is more representative of typical wood flour grain size) yielded a 75% increase in tensile strength for the 29% cane pulp blend. The 69% pulp blend performed the same as the pure resin case, although it exhibited a 40% better tensile strength at the 80–170 mesh level. It was postulated in this study that a higher level of cane fibers interfered with the phenolic wetting of the cane, which resulted in weaker fiber/matrix bonding. Although the cane apparently added significant strength to the phenolic, it should be noted that the pure phenolic resin was not particularly strong; its tensile strength was only 16.4 MPa (2380 psi). This may have allowed for a greater level of reinforcement from any filler.

A recent study reported that corn-based DDGS concentrations of 25–50% represent reasonable inclusion values with phenolic resin as sufficient mechanical strength is still available in the finished blends (Tatara et al., 2007). However, the study was preliminary in nature and relied upon few data points; also, no physical properties were measured. On the other hand, the objective of this study is to pursue a much more systematic, detailed, and complete examination of this novel bio-filled resin by measuring several mechanical and physical properties of DDGS-based blends.

2. Experimental conditions

To develop design data, the blend composition, molding pressure, and molding temperature were varied. The DDGS content included 0% (pure resin), 25%, 50%, and 75%. The molding pressure was set to 13.8, 34.5, or 48.3 MPa (1.0, 2.5, or 3.5 tons/in.²) while the temperatures tested were 157 °C (315 F), 174 °C (345 F), or 191 °C (375 F). A design of experiments approach was used to systematically evaluate the effects of each parameter, using a 3 × 3 × 3 (for a total of 27 treatment combinations) factorial design which was implemented as a Completely Randomized Design. For every combination of DDGS concentration, pressure, and temperature, three test specimens (i.e., $n=3$ for each treatment combination) were molded and tested to quantify mechanical properties (i.e.,

tensile yield strength, Young's modulus, percent elongation, and hardness), as well as physical and performance properties (i.e., density, water absorption, and biodegradability) at that set of conditions. The mechanical properties were obtained through tensile testing per ASTM D638-03 with yield strength calculated using original specimen cross-sectional area. Also, consistent with engineering strain, the change in length of the specimen's portion between the tensile grips was divided by its original length then converted to a percent basis for elongations. The mass density was measured via ASTM D792-00, and biodegradation studies followed ASTM D5988-03 and ASTM D6400-04. Water absorption was evaluated following ASTM D570-98, using 2 h, 24 h, and 1 week of immersion. Surface hardness was measured with a Durometer-type Shore D scale indenter; each specimen was sampled by averaging eight readings at various locations.

Mold construction, instrumentation, and data acquisition, as well as molding procedures, were consistent with previous work (Tatara et al., 2007). Additionally, for the present effort, a three-cavity compression mold (Fig. 1) was created to produce multiple test specimens. The mold was made from 7075 T651 aluminum. It has a mass of 5.8 kg (12.75 lb_m) and, when closed, measures 191 mm (7.50 in.) in length, 152 mm (6.00 in.) in width, and 70 mm (2.75 in.) in height. The molder was a commercial, 267 kN (30-ton) electric/hydraulic unit with 305 mm × 305 mm (12 in. × 12 in.) heated platens. Tensile bar samples were 165.1 mm (6.5 in.) long, with width and thickness

in the narrowed (i.e., break) region measuring 12.7 mm (0.5 in.) and 3.2 mm (0.125 in.), respectively. Consistent samples were produced, as demonstrated by their uniform thickness, even when molding up to three at once.

The phenol and formaldehyde resin was a commercial grade powder consisting of 91.5% phenolic with 8.5% hexa curing agent without any other additives or fillers as found in conventional molding compounds. The distillers dried grains with solubles were obtained from a commercial dry-grind ethanol plant, and had a 12.3% moisture content (on a dry basis), a protein content of 27.6%, a fiber content of 11.1%, a lipid content of 9.3%, an ash content of 4.2%, and other carbohydrates of 47.8%. The DDGS was utilized in its raw, untreated form. The two materials were thoroughly mixed by stirring, poured into the bottom half of the mold, and compression molded at the desired pressure and temperature levels; depending on pressure level used, more than one bar could be molded simultaneously. All test specimens were produced with the same molding procedure, which began with a cold mold. Each compression molding cycle required about 30 min to bring the mold from room temperature up to the set conditions. After a given molding time, another 15 min were necessary to cool the mold, during which the pressure was reduced to 8.8 MPa (0.64 tons/in.²), and remove the tensile bar specimen(s). Table 1 presents a sample pressure and temperature cycle.

The resin and biofiller powders were thoroughly mixed without any chemical reaction or treatment prior to the compression molding process. Chemically combining a biofiller with phenolic, then molding, is a more complex activity, but would impart added strength to molded specimens (Maldas and Shiraishi, 1996, 1997) and should be the focus of a future investigation. But the aim of this study was to promote the use of the corn-based DDGS available directly from an ethanol plant. Any pretreatments (including drying) make the DDGS less viable as low-cost filler.



Fig. 1 – Three-cavity, aluminum compression mold constructed for the study; units of the scale bar are centimeters.

3. Statistical analysis of property data

3.1. Statistical technique

Upon molding and testing the specimens, property data were then analyzed with the Proc GLM procedure (SAS V.8, SAS Institute, Cary, NC) to identify significant main and interaction effects at an α level (Type I error rate) of 0.05. Mean values associated with significant effects were compared using least significant differences (LSD). Additionally, property relationships were examined by Pearson product-moment correlation analysis.

3.2. Property relationships

Relationships between all measured mechanical, physical, and performance properties were investigated using correlation analysis. Thirty-two of the resulting 66 Pearson product-moment correlations were significant ($p < 0.05$); the remainder of the correlations were not. The correlation coefficient quantifies the strength of the linear relationship between two variables, and eight of the variable combinations

Table 1 – Sample molding parameters over entire molding cycle

Time (min)	Molding pressure MPa (tons/in. ²)	Upper platen temperature		Lower platen temperature		Mold temperature in lower half		Mold temperature in upper half	
		°C	(F)	°C	(F)	°C	(F)	°C	(F)
0	48.5 (3.52)	27	(80)	27	(80)	24	(76)	27	(80)
5	45.9 (3.33)	77	(170)	66	(150)	56	(133)	55	(131)
10	47.6 (3.45)	96	(205)	88	(190)	78	(172)	78	(172)
15	47.6 (3.45)	122	(252)	106	(223)	99	(211)	103	(218)
20	47.6 (3.45)	154	(310)	133	(272)	122	(252)	129	(264)
25	48.5 (3.52)	167	(332)	160	(320)	141	(286)	146	(295)
30	48.5 (3.52)	199	(390)	196	(385)	172	(342)	179	(354)
31	8.8 (0.64)	Platen		Heaters		Turned		Off.	
40	7.9 (0.58)	34	(93)	27	(80)	54	(130)	107	(225)

Table 2 – Effect of DDGS content on resulting strength properties (\pm one standard deviation)

DDGS (%)	Yield strength (MPa (psi))	Break strength (MPa (psi))	Young's modulus (MPa (psi))	Elongation at yield (%)	Elongation at break (%)
0	28.5 (4142) \pm 22%	28.5 (4142) \pm 22%	2632 (382,000) \pm 12%	1.13 \pm 27%	1.13 \pm 27%
25	14.5 (2102) \pm 14%	14.6 (2118) \pm 12%	2296 (333,000) \pm 10%	0.65 \pm 17%	0.65 \pm 16%
50	6.7 (969) \pm 17%	7.6 (1105) \pm 15%	1379 (200,000) \pm 16%	0.50 \pm 23%	0.59 \pm 21%
75	4.3 (621) \pm 17%	5.6 (814) \pm 23%	841 (122,000) \pm 27%	0.54 \pm 21%	0.85 \pm 19%

had resulting correlation coefficients greater than $|0.80|$, while four (2 h absorption \times 24 h absorption; 2 h absorption \times 1 week absorption; 24 h absorption \times 1 week absorption; DDGS level \times Young's modulus) had coefficients greater than $|0.90|$, and thus exhibited fairly strong linear relationships. Some of these correlations were expected prior to analysis. All of the water absorption indices (i.e., 2 h, 24 h, 1 week) were related, which was anticipated *a priori*. More importantly, DDGS level was related to most of the measured properties, which was reflective of the results shown in Figs. 3–5, Tables 2 and 3. Additionally, the mechanical properties for the resulting phenolic with DDGS were related to the physical and performance properties, which was directly due to the influence of DDGS level on all of the resulting properties.

4. Effects of introducing DDGS to phenolic resin

Examining the effects due to the experimental factors (Table 2) indicates that DDGS level has a significant effect on all mechanical properties studied. Pressure, on the other hand, does not have an effect on any of the properties, and for the most part, the effects of temperature are not significant either (except for Young's modulus). Regarding physical and perfor-

mance properties (Table 3), DDGS level has a significant effect on water absorption behavior as well as biodegradability and hardness. Pressure and temperature, on the other hand, have little effect on these properties. Comparisons were also made with the pure resin (i.e., baseline) data. Although all values were established from an arithmetic mean of three specimens taken at each combination of DDGS concentration, pressure, and temperature, it should be noted that there was substantial scatter in many of the individual data points; Tables 2 and 3 include the standard deviation range for each averaged measurement. Even so, several trends in the data are evident; these will be discussed in detail.

4.1. Mechanical properties

In the 0% and 25% DDGS cases, the resulting materials are brittle, with yield and break occurring simultaneously, with little to no deviation from the proportional limit. Thus, traditional yield, where the material elongates without a corresponding increase in tension, did not occur. However, at the 50% and 75% DDGS levels, there was some actual yielding and observed ductility (but not in every case). The proportional limit, yield point, and break tensile could be distinguished, and the break strength was 15% and 30% greater than the proportional limit

Table 3 – Effect of DDGS content on other properties (\pm one standard deviation)

DDGS (%)	Biodegradability (mass% degraded)	Durometer hardness (Shore D)	Density (g/cm ³ (lb _m /ft ³))	Water absorption (mass%)		
				2 h	24 h	1 week
0	0.0 \pm 0%	93 \pm 2%	1.20 (74.9) \pm 52%	0.1 \pm 0%	0.2 \pm 0%	0.5 \pm 0%
25	8.6 \pm 12%	82 \pm 2%	1.24 (77.4) \pm 15%	1.6 \pm 1%	2.8 \pm 3%	7.9 \pm 8%
50	24.4 \pm 10%	72 \pm 3%	1.23 (76.8) \pm 35%	5.0 \pm 2%	13.2 \pm 5%	29.4 \pm 9%
75	38.4 \pm 6%	68 \pm 3%	1.22 (76.2) \pm 41%	6.2 \pm 3%	15.9 \pm 8%	33.5 \pm 17%

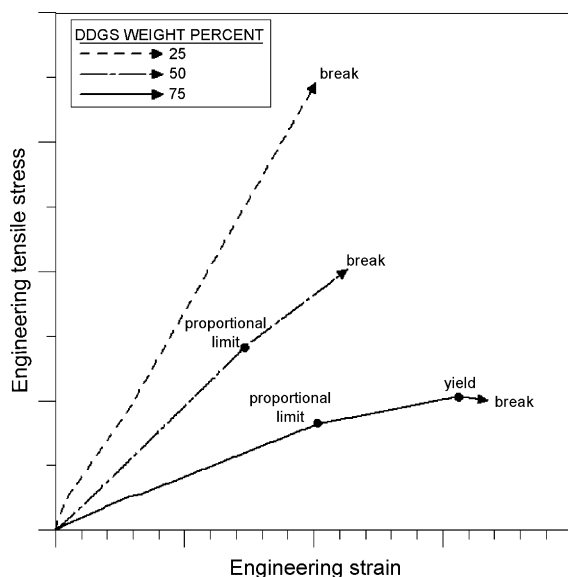


Fig. 2 – Typical stress–strain curves for DDGS/phenolic blends.

for 50% and 75% DDGS, respectively, as noted in Table 2. (Likewise, the break elongation value was 20% and 60% larger for 50% and 75% DDGS, respectively, than the proportional limit extension.) This behavior is illustrated in Fig. 2, which qualitatively presents the three types of stress–strain curves generally encountered in this study. Nevertheless, for the purposes of comparison, the tensile yield strengths reported here (and the corresponding percent elongations) represent the initial deviation from linearity, or the proportional limit, if the sample did not truly yield before fracture. This approach is justified because it was observed that the proportional limit represented permanent, inelastic breaking of bonds in the test specimens. Thus, the reported strength values represent the elastic limit of the materials, and are the pertinent quantities for design. Along these lines, the reported tensile yield corresponds to the break, proportional limit, and proportional limit (as labeled in Fig. 2) for the 25%, 50%, and 75% DDGS levels, respectively. Also, the Young's modulus was taken as the initial modulus (low load conditions) even though above a DDGS level of 25% showed secondary, less rigid linearity after the proportional limit was reached; this is particularly noted in the 50% filled specimens. It is postulated that this added ductility is from the fibrous DDGS bearing proportionally more of the tensile loading.

The results from this study are best presented by the ratio of a given property value for any DDGS blend, divided by the value of that property for the pure resin (i.e., 0% DDGS) case. This is shown in Fig. 3, which plots tensile yield strength, Young's modulus, and percent elongation at yield. The ratios are a direct measure of the property change as DDGS is added at greater inclusion levels. The average baseline, or pure resin case, data are 28.5 MPa (4142 psi), 2632 MPa (382,000 psi), and 1.13% for the tensile yield, Young's modulus, and percent elongation at yield, respectively. At 25% DDGS, the tensile yield strength is approximately half that of the pure resin case. Higher levels of biofiller further reduce the strength to nearly

one-quarter (at 50% DDGS), and under one-sixth (at 75% DDGS) the baseline. Young's modulus is also reduced from that of 100% resin, but less severely. At 25% DDGS, a 10–15% stiffness reduction is noted, while 50% and 75% filler levels cause 50% and 70% decreases in Young's modulus, respectively. The ductility of the blends, as measured through percent elongation, is restricted about 50% by inclusion of biofiller, although a clear trend is not seen. The percent elongation at yield is actually better at 75% DDGS than at 50%. It is possible that higher DDGS content begins to contribute more to the elongation than the resin matrix. Fig. 3 also displays standard deviation ranges for the tensile yield and modulus (other property uncertainty ranges are omitted to maintain figure clarity); all exact values, including those for elongation and hardness, are available in Tables 2 and 3. Additionally, surface hardness trending is presented in Fig. 4 and indicates a general softening as DDGS increases; the Shore D values are about 10% and 25% lower for 25% DDGS and 50–75% DDGS, respectively.

Fig. 3 provides results from some other studies for comparison purposes. It should be noted that direct data comparison is difficult, due to varying molding conditions, blends, and lack of pure resin data in many studies. Without pure resin data, the relative effects of adding biofiller cannot be ascertained. Generally, tensile strength ratios from this study are better than lignin blends (Kharade and Kale, 1998). Sugar cane biofiller strength ratios were significantly greater than the DDGS-filled blends; but, as already discussed, their pure phenolic matrix was not very strong initially (Leite et al., 2004). Likewise, in Fig. 4, hardness reduction in the DDGS blends is consistent with that observed by Hattali et al. (2005) for Alpha grass lignin and wood flour composites.

4.2. Physical properties

Regarding physical properties, without biofiller the resin has negligible water absorption (Table 3). For the 25% DDGS case,

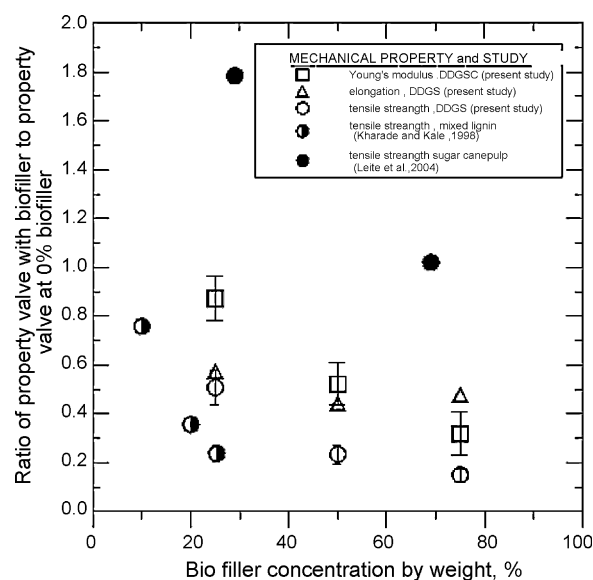


Fig. 3 – Comparison of mechanical properties, as a function of biofiller concentration, in phenolic resin-based blends. Error bars represent \pm one standard deviation.

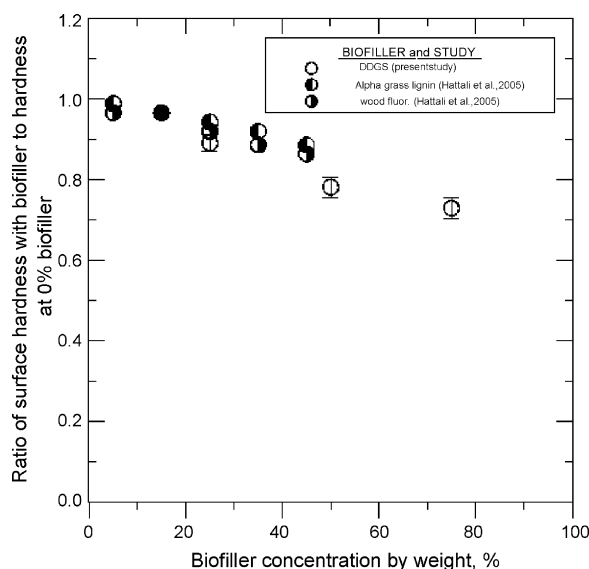


Fig. 4 – Comparison of surface hardness, as a function of biofiller concentration, in phenolic resin-based blends. Error bars represent \pm one standard deviation.

water absorption ranged from 1.6% to 7.9% as the immersion time ranged from 2 h to 1 week. The uptake increased for the 50% blend, up to 30% after 1 week. The 75% DDGS case represented a slight increase in water absorption compared to that at 50%. Overall, the presence of DDGS creates considerable porosity in the material. This is in close agreement with 24-h immersion sugar cane pulp/phenolic blend data of Leite et al. (2004), also displayed in Fig. 5; the difference is somewhat higher water absorption for the 50% and 75% DDGS cases compared to 69% cane pulp. Fig. 5 displays standard deviation ranges for the 50% and 75% blends where the deviation is significant; note that the uncertainty increases with DDGS content and immersion time.

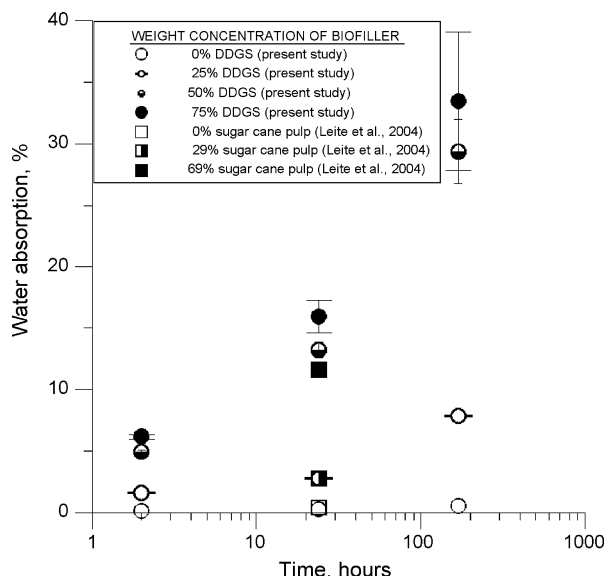


Fig. 5 – Water absorption for biofiller/phenolic blends. Error bars represent \pm one standard deviation.

The DDGS biofiller also added weight and improved biodegradability to the molded blends. Density increases with DDGS; values for the molded specimens were 1.20, 1.24, 1.23, and 1.22 g/cm³ (74.9, 77.4, 76.8, and 76.2 lb_m/ft³) for 0%, 25%, 50%, and 75% DDGS, respectively. The values near 1.2 g/cm³ (74.9 lb_m/ft³) are comparable to those for sugar cane/phenolic blends where densities were 1.24 g/cm³ (77.4 lb_m/ft³) and 1.15 g/cm³ (71.8 lb_m/ft³) at 29% and 69% cane pulp (35–80 mesh size), respectively (Leite et al., 2004). The presence of biofiller does offer biodegradability (noted as mass% degraded in Table 3) where none had previously existed (pure resin baseline). A 9% biodegradability is observed with 25% DDGS, 25% with 50% DDGS, and 40% with 75% DDGS. Certainly, the rather elevated water absorption of the blends contributed to the significant biodegradability of the thermoset-based material.

4.3. Effects of molding parameters on DDGS blends

Other than varying the DDGS content, the molding variables of pressure and temperature were also examined. The molding pressures were 13.8, 34.5, or 48.3 MPa (1.0, 2.5, or 3.5 tons/in.²) while the temperatures tested were 157 °C (315 F), 174 °C (345 F), or 191 °C (375 F). Generally, molding pressure and temperature did not have a significant effect on tensile strength. The combination of higher temperature and pressure did seem to slightly improve tensile yield strength for 25% DDGS. However, the 75% level was negatively affected by raising the molding temperature from 157 °C (315 F) to 191 °C (375 F), as one-quarter of the strength was lost. In the case of elongation at yield, no discernable pressure trends were found, although it did somewhat improve as molding temperature increased. Increasing molding temperature somewhat decreased the Young's modulus especially for 75% DDGS. At 191 °C (375 F), increasing the pressure did stiffen the test specimens when the DDGS content was at least 50%; otherwise pressure effects were negligible.

Raising the molding pressure and temperature did slightly reduce the water absorptivity but the correlation was not strong. Similarly, an increase in molding temperature resulted in less dense specimens, especially above 174 °C (345 F), while pressure did not consistently trend. Surface hardness and biodegradability are not dependent on either pressure or molding temperature.

Generally, in the ranges tested, molding conditions far less affected the mechanical and physical properties of the blends. Quantitative details concerning the statistical analyses and correlation results for any trends among the molding parameters and material properties are available elsewhere (Suraparaju, 2007). Clearly, the DDGS content most strongly influenced the final properties.

5. Conclusions

In general, the data are in the range of other studies that have examined biofillers, and conditions have been quantified to successfully produce this unique bio-filled resin. The available mechanical and physical properties are of use for potential designs utilizing this material. A 25% DDGS concentration

retains sufficient strength and has limited water absorption compared to elevated DDGS blends. Molding pressure can range from 13.8 to 48.3 MPa (1.0–3.5 tons/in.²), and 174°C (345 F) is an effective molding temperature. Further development and characterization of this material will provide data that are essential for the design of equipment and processing facilities, as well as optimization of subsequent unit operations in production settings.

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